

What is Claimed is:

- 1 1. A process for the manufacture of a LiMPO_4 powder, comprising the steps
2 of:
3 providing an equimolar aqueous solution of Li^{1+} , M^{n+} , and PO_4^{3-} prepared
4 by dissolving components which are susceptible to coexist as solutes in an aqueous
5 system and which, upon heating at a temperature below 500°C , decompose to form a
6 pure homogeneous Li and M phosphate precursor;
7 evaporating water from the solution, thereby producing a solid mixture;
8 decomposing the solid mixture at a temperature below 500°C to form a
9 pure homogeneous Li and M phosphate precursor; and
10 annealing the precursor at a temperature of less than 800°C in an inert or
11 reducing atmosphere, thereby forming a LiMPO_4 powder;
12 wherein M^{n+} is one or more of Fe^{2+} , Fe^{3+} , Co^{2+} , Ni^{2+} , and Mn^{2+} , and M is
13 $\text{Fe}_x\text{Co}_y\text{Ni}_z\text{Mn}_w$, with $0 \leq x \leq 1$, $0 \leq y \leq 1$, $0 \leq z \leq 1$, $0 \leq w \leq 1$, and $x + y + z + w = 1$.
- 1 2. The process according to claim 1, wherein in the step of annealing the
2 precursor, the annealing temperature is less than 600°C .
- 1 3. A process for the manufacture of a LiFePO_4 powder, comprising the
2 steps of:
3 providing an equimolar aqueous solution of Li^{1+} , Fe^{3+} , and PO_4^{3-} prepared
4 by dissolving components which are susceptible to coexist as solutes in an aqueous
5 system and which, upon heating at a temperature below 500°C , decompose to form a
6 pure homogeneous Li and Fe phosphate precursor;
7 evaporating water from the solution, thereby producing a solid mixture;
8 decomposing the solid mixture at a temperature below 500°C to form a
9 pure homogeneous Li and Fe phosphate precursor; and
10 annealing the precursor at a temperature of less than 800°C in a reducing
11 atmosphere, thereby forming a LiFePO_4 powder.
- 1 4. The process according to claim 3, wherein in the step of annealing the
2 precursor, the annealing temperature is less than 600°C .
- 1 5. The process according to claims 3, wherein the Fe^{3+} bearing component
2 is iron nitrate.

1 6. A powder for use in lithium insertion-type electrodes with a formula
2 LiMPO_4 having an average particle size of less than $1\mu\text{m}$, wherein M is $\text{Fe}_x\text{Co}_y\text{Ni}_z\text{Mn}_w$,
3 with $0 \leq x \leq 1$, $0 \leq y \leq 1$, $0 \leq z \leq 1$, $0 \leq w \leq 1$, $x + z + w > 0$, and $x + y + z + w = 1$.

1 7. The powder according to claim 6, wherein M is Fe, the powder having a
2 reversible electrode capacity of at least 65% of a theoretical capacity when used as an
3 active component in a cathode that is cycled between 2.70 and 4.15 V vs. Li^+/Li at a
4 discharge rate of C/5 at 25°C .

1 8. A powder for use in lithium insertion-type electrodes prepared by a
2 process comprising the steps of:
3 providing an equimolar aqueous solution of Li^{1+} , M^{n+} , and PO_4^{3-} prepared
4 by dissolving components which are susceptible to coexist as solutes in an aqueous
5 system and which, upon heating at a temperature below 500°C , decompose to form a
6 pure homogeneous Li and M phosphate precursor;
7 evaporating water from the solution, thereby producing a solid mixture;
8 decomposing the solid mixture at a temperature below 500°C to form a
9 pure homogeneous Li and M phosphate precursor; and
10 annealing the precursor at a temperature of less than 600°C in an inert or
11 reducing atmosphere, thereby forming a LiMPO_4 powder;
12 wherein M^{n+} is one or more of Fe^{2+} , Fe^{3+} , Co^{2+} , Ni^{2+} , and Mn^{2+} , and M is
13 $\text{Fe}_x\text{Co}_y\text{Ni}_z\text{Mn}_w$, with $0 \leq x \leq 1$, $0 \leq y \leq 1$, $0 \leq z \leq 1$, $0 \leq w \leq 1$, and $x + y + z + w = 1$.

1 9. The powder according to claim 8, wherein M^{n+} is Fe^{3+} , M is Fe, and the
2 annealing occurs in a reducing atmosphere.

1 10. A battery comprising a lithium insertion-type electrode including a
2 powder prepared by a process comprising the steps of:
3 providing an equimolar aqueous solution of Li^{1+} , M^{n+} , and PO_4^{3-} prepared
4 by dissolving components which are susceptible to coexist as solutes in an aqueous
5 system and which, upon heating at a temperature below 500°C , decompose to form a
6 pure homogeneous Li and M phosphate precursor;
7 evaporating water from the solution, thereby producing a solid mixture;
8 decomposing the solid mixture at a temperature below 500°C to form a
9 pure homogeneous Li and M phosphate precursor; and

10 annealing the precursor at a temperature of less than 600° C in an inert or
 11 reducing atmosphere, thereby forming a LiMPO_4 powder;
 12 wherein M^{n+} is one or more of Fe^{2+} , Fe^{3+} , Co^{2+} , Ni^{2+} , and Mn^{2+} , and M is
 13 $\text{Fe}_x\text{Co}_y\text{Ni}_z\text{Mn}_w$, with $0 \leq x \leq 1$, $0 \leq y \leq 1$, $0 \leq z \leq 1$, $0 \leq w \leq 1$, and $x + y + z + w = 1$.

1 11. The battery according to claim 10, wherein the powder has an average
 2 particle size of less than $1\mu\text{m}$ and $x + z + w > 0$.

1 12. The battery according to claim 11, wherein M is Fe, the powder having a
 2 reversible electrode capacity of at least 65% of a theoretical capacity when used as an
 3 active component in a cathode that is cycled between 2.70 and 4.15 V vs. Li^+/Li at a
 4 discharge rate of C/5 at 25° C.

1 13. The battery according to claim 10, wherein M^{n+} is Fe^{3+} , M is Fe, and the
 2 annealing occurs in a reducing atmosphere.

1 14. A process for the manufacture of a lithium insertion-type electrode
 2 comprising the steps of:
 3 providing an equimolar aqueous solution of Li^{1+} , M^{n+} , and PO_4^{3-} prepared
 4 by dissolving components which are susceptible to coexist as solutes in an aqueous
 5 system and which, upon heating at a temperature below 500° C, decompose to form a
 6 pure homogeneous Li and M phosphate precursor;
 7 evaporating water from the solution, thereby producing a solid mixture;
 8 decomposing the solid mixture at a temperature below 500° C to form a
 9 pure homogeneous Li and M phosphate precursor;
 10 annealing the precursor at a temperature of less than 600° C in an inert or
 11 reducing atmosphere, thereby forming a LiMPO_4 powder;
 12 providing a mixture of the LiMPO_4 powder and a conductive carbon
 13 bearing powder; and
 14 milling the mixture during a period of time to optimize a reversible
 15 electrode capacity of the electrode;
 16 wherein M^{n+} is one or more of Fe^{2+} , Fe^{3+} , Co^{2+} , Ni^{2+} , and Mn^{2+} , and M is
 17 $\text{Fe}_x\text{Co}_y\text{Ni}_z\text{Mn}_w$, with $0 \leq x \leq 1$, $0 \leq y \leq 1$, $0 \leq z \leq 1$, $0 \leq w \leq 1$, and $x + y + z + w = 1$.

1 15. The process according to claim 14, wherein M is Fe, the conductive
2 carbon powder is one of Acetylene Black and Carbon Super P, the weight ratio of
3 LiFePO_4 /carbon is between 75/25 and 85/15, and the milling time is between 15 and 25
4 minutes.

1 16. The process according to claim 14, wherein the powder has an average
2 particle size of less than $1\mu\text{m}$ and $x + z + w > 0$.

1 17. The process according to claim 16, wherein M is Fe and the reversible
2 electrode capacity is at least 65% of a theoretical capacity when used as an active
3 component in a cathode that is cycled between 2.70 and 4.15 V vs. Li^+/Li at a discharge
4 rate of C/5 at 25°C .